# PATENT ABSTRACTS OF JAPAN

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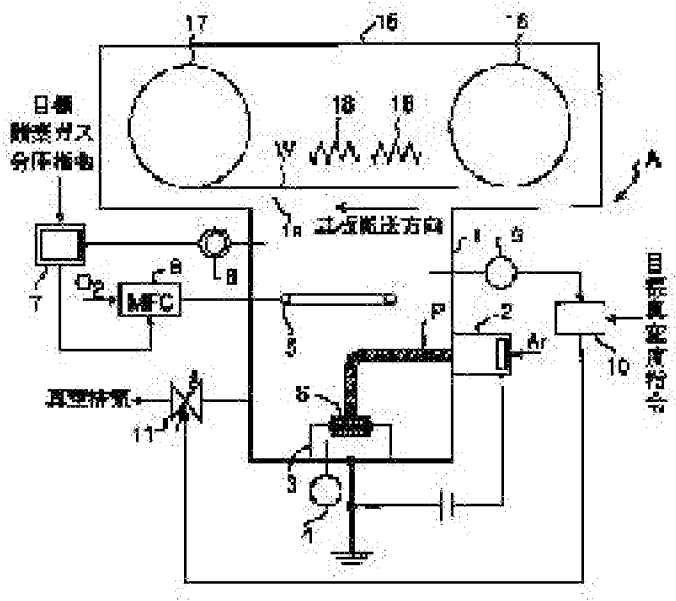
## (54) ACTIVATING REACTION VAPOR DEPOSITION METHOD FOR OXIDE THIN FILM

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a activating reaction vapor depositing method for oxide thin film capable of stably obtaining thin film over a long time.

SOLUTION: In a activating reaction vapor depositing method in which a pressure gradient type plasma gun 2 is used to form oxide thin film on a substrate W, the degree of vacuum in a treating chamber is detected, and the exhausting rate from the treating chamber is controlled so as to make the detected value equal to the set value. Furthermore, the partial pressure of oxygen in the treating chamber is detected, and the amt. of oxygen to be introduced into the treating chamber is controlled so as to make the detected value equal to the set value.



* NOTICES *



## DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention relates to the membrane formation controlling method at the time of forming an oxide film to a substrate by the activated reactive evaporation of oxide films, such as an ITO film and a MgO film, and the activated reactive evaporation which uses a pressure gradient type plasma gun in detail.
[0002]
[Description of the Prior Art]As for the ITO film used for a liquid crystal or a touch panel, and the MgO film used for an AC type plasma display, the demand of highly-efficient-izing is increasing with the spread.
Furthermore,-izing from Sadao Takayasu is desired strongly.
In order to form oxide films, such as an ITO film and a MgO film, to a substrate etc. conventionally, the forming-membranes method by dry processes, such as the vacuum deposition method and sputtering process using an electron beam, or activated reactive evaporation which uses a pressure gradient type plasma gun, is generally performed. Under the present circumstances, in order to complement the oxygen which secedes from an oxide deposition material or its evaporation particle or to adjust membraneous quality during membrane formation, while introducing a constant rate of oxygen into the membrane formation interior of a room, The opening of the valve for pressure control in which the exhaust speed by a vacuum pump was provided at the exhaust-port side is adjusted, and it is made to make regularity membrane formation room pressure power under membrane formation for the purpose of stabilizing more the degree of crystallinity and crystal orientation of the formed thin film.
[0003]
[Problem(s) to be Solved by the Invention]However, in the case where a constant rate of oxygen is introduced in order to complement the oxygen which secedes from an oxide deposition material or its evaporation particle or to adjust membraneous quality during membrane formation, By change of the amount of oxygen from which it secedes from adsorption or every place to every place of the oxide deposition material itself and a membrane formation indoor part, the amount of oxygen of the membrane formation indoor part under membrane formation will be changed, as a result, the oxygen tension of the membrane formation interior of a room will be changed, and it had the problem that it was difficult to obtain the stable film performance. For example, in membrane formation of ITO, change of the oxygen tension of the membrane formation interior of a room knows changing the specific resistance and

transmissivity of the formed film widely. In membrane formation of MgO, change of the oxygen tension of the membrane formation interior of a room also knows changing the degree of crystallinity and crystal orientation of the formed film. A constant rate of oxygen is supplied for prolonged membrane formation to the membrane formation interior of a room, moving the crucible with which the oxide deposition material was especially installed using the pressure gradient type plasma gun, And in the case where do not control membrane formation room pressure power, but it carries out, Since it is not only difficult to obtain the film performance which the oxygen tension of the membrane formation interior of a room was not stabilized at as shown in drawing 2, but was therefore stabilized since change of the amount of oxygen of a membrane formation indoor part is remarkable, but the pressure of a membrane formation room was remarkably changed as shown in drawing 3, knowledge that membrane formation speed is also changed sharply was acquired. Also in the case where it controls by adjusting the opening of the valve for pressure control in which the exhaust speed by a vacuum pump was provided at the exhaust-port side so that the membrane formation room pressure power under membrane formation may become fixed, Since change of the membrane formation room pressure power under membrane formation did not originate only in change of oxygen tension, as shown in drawing 4, the oxygen tension of the membrane formation interior of a room will be changed, and it had the problem that it was difficult to obtain the stable film performance. This invention solves said problem and it aims at providing the membrane formation controlling method of an oxide film for the ability to obtain the film performance stable over the long time.

[0004]

[Means for Solving the Problem]In activated reactive evaporation which forms an oxidation thin film in a substrate using a pressure gradient type plasma gun in order that this invention may attain said purpose, A degree of vacuum in a processing chamber is detected, and oxygen tension in a processing chamber is detected, and while controlling an exhaust speed from a processing chamber so that this detection value becomes equal to a preset value, an oxygen introduction amount into a processing chamber is controlled so that this detection value becomes equal to a preset value.

[0005]

[Embodiment of the Invention]Below, an embodiment of the invention is described according to drawing 1. In the figure, the film deposition system A consists of the 1st processing chamber 1 which does heating evaporation of the deposition material S, and the 2nd processing chamber 15 which accommodates a substrate, the side attachment wall of said 1st processing chamber 1 is equipped with the publicly known pressure gradient type plasma gun 2, and the crucible 3 which stored the deposition material S in the upper part is installed in the pars basilaris ossis occipitalis. And this crucible 3 carries out reciprocation moving of the predetermined section with the drive 4 from the lower stream to the upper stream to the lower stream from the transportation direction upper stream of film like substrate W which carries out the following. The gaseous oxygen supply nozzle 5 is formed in said 1st processing chamber 1, The oxygen gas partial pressure in the 1st processing chamber 1 is measured with the mass spectrometer 6, and a difference with the target oxygen gas partial pressure instructions beforehand set to the oxygen gas part pressure signal is calculated with the oxygen gas flow rate operation machine 7, Control supply is carried out into the 1st processing chamber 1 from the oxygen gas of the specified quantity from said gaseous oxygen supply nozzle 5 so that the oxygen gas partial pressure in the 1st processing chamber 1 may serve as a predetermined value with the massflow controller 8.

[0006]The degree of vacuum in the 1st processing chamber 1 is measured with the vacuum meter 9, and

this degree-of-vacuum signal is inputted into the pressure computing unit 10, It is compared with the target-degree-of-vacuum instructions set up beforehand here, the pressure control valve 11 operates based on the difference, and the degree of vacuum in the 1st processing chamber 1 is controlled to a predetermined value.

[0007]Said 2nd processing chamber 15 is what was provided via the opening 1a above said 1st processing chamber 1, and while the delivery roll 16 and the winding roll 17 of film like substrate W are allocated inside this 2nd processing chamber 15, the heater 18 is allocated among both the rolls 16 and 17.

[0008]In the above composition, said pressure gradient type plasma gun 2 is operated, and the plasma beam P is converged on the deposition material S of the crucible 3. A lot of deposition materials S are stored so that it can be adapted for prolonged membrane formation, and predetermined section reciprocation moving of the crucible 3 is continuously carried out from the lower stream to the upper stream with the drive 4 in the lower stream from the direction-of-movement upper stream of conveyance of film like substrate W. And the deposition material S which heated and evaporated in said plasma beam P reacts also to the oxygen gas supplied from the gaseous oxygen supply nozzle 5, making it secede from a part of own oxygen in this process, and forms an oxide film in film like substrate W. Under the present circumstances, the mass spectrometer 6 always measures the oxygen gas partial pressure in said 1st processing chamber 1, and sends that oxygen gas part pressure signal to said oxygen gas flow rate operation machine 7. The oxygen gas amount of supply is computed by calculating the difference of the target oxygen gas partial pressure instructions and the oxygen gas part pressure signal from the mass spectrometer 6 which are beforehand set to the oxygen gas flow rate operation machine 7, Outputting to the massflow controller 8 by making this into an oxygen gas amount-of-supply signal, the massflow controller 8 concerned supplies that amount of supply to the gaseous oxygen supply nozzle 5 stably in response to this oxygen gas amount-of-supply signal.

[0009]On the other hand, the vacuum meter 9 always measures the degree of vacuum in said 1st processing chamber 1, and sends the degree-of-vacuum signal to said pressure computing unit 10. The pressure computing unit 10 concerned computes a valve opening signal by calculating the difference of the target-degree-of-vacuum instructions and the degree-of-vacuum signal from the vacuum meter 9 which are set up beforehand, and outputs this to the pressure control valve 11. And the pressure control valve 11 concerned operates in response to this valve opening signal, and leads the degree of vacuum in said 1st processing chamber 1 to a desired value.

[0010]

[Effect of the Invention]In order to measure separately the oxygen gas partial pressure and the degree of vacuum in the 1st processing chamber and to control them by the above explanation clearly according to this invention, Even if it changes the amount of secession under membrane formation of the oxygen which stuck to every place of the amount of oxygen, or a membrane formation indoor part which secedes from the oxide deposition material itself and evaporation particles, During membrane formation, since it becomes constant [ the oxygen gas partial pressure in said 1st processing chamber ] and becomes still always more nearly constant [ the pressure in said 1st processing chamber ] during membrane formation, the film performance stable over the long time can always be obtained, without also generating change of membrane formation speed.

[Translation done.]

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審査請求　未請求　請求項の数１　ＯＬ　（全　４　頁）

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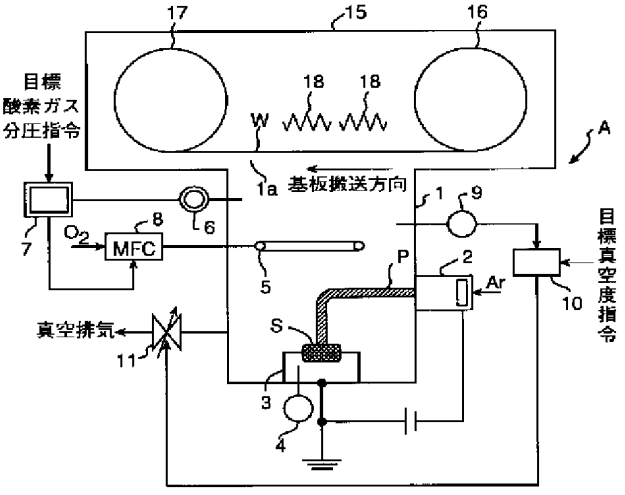
(54)【発明の名称】　酸化物薄膜の活性化反応蒸着法

(57)【要約】
【課題】　長時間にわたって安定した薄膜を得る酸化物薄膜の活性化反応蒸着法を提供する。
【解決手段】　圧力勾配型プラズマガン２を使用して基板Ｗに酸化薄膜を形成する活性化反応蒸着法において、処理室内の真空度を検出し、この検出値が設定値と等しくなるように処理室からの排気速度を制御する。また、処理室内の素分圧を検出し、この検出値が設定値と等しくなるように処理室内への酸素導入量を制御する。



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【特許請求の範囲】

【請求項１】　圧力勾配型プラズマガンを使用して基板に酸化薄膜を形成する活性化反応蒸着法において、処理室内の真空度を検出し、この検出値が設定値と等しくなるように処理室からの排気速度を制御する一方、処理室内の酸素分圧を検出し、この検出値が設定値と等しくなるように処理室内への酸素導入量を制御することを特徴とする酸化物薄膜の活性化反応蒸着法。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は、ＩＴＯ膜やＭｇＯ膜などの酸化物薄膜の活性化反応蒸着法、詳しくは圧力勾配型プラズマガンを使用する活性化反応蒸着法により基板に酸化物膜を成膜する際の成膜制御法に関する。

【０００２】

【従来の技術】液晶あるいはタッチパネル等に使用されるＩＴＯ膜や、ＡＣ型プラズマディスプレイに使用されるＭｇＯ膜は、その普及に伴って高性能化の要求が高まっており、さらには高安定生産化が強く望まれている。従来、ＩＴＯ膜やＭｇＯ膜などの酸化物薄膜を基板等に成膜するには、電子ビームを利用した真空蒸着法、スパッタリング法、あるいは圧力勾配型プラズマガンを使用する活性化反応蒸着法などのドライプロセスによる成膜法が一般的に行なわれている。この際、成膜中に酸化物蒸着材料やその蒸発粒子から離脱する酸素を補完したり、膜質を調整するために一定量の酸素を成膜室内に導入するとともに、成膜された薄膜の結晶化度や結晶方位をより安定化させる目的で、真空ポンプによる排気速度をその排気口側に設けられた圧力制御用バルブの開度を調整し、成膜中の成膜室圧力を一定にするようにしている。

【０００３】

【発明が解決しようとする課題】しかしながら、成膜中に酸化物蒸着材料やその蒸発粒子から離脱する酸素を補完したり、膜質を調整するために一定量の酸素を導入する場合においては、酸化物蒸着材料自身や成膜室内部の各所に吸着または各所より離脱する酸素量の変動により、成膜中における成膜室内部の酸素量が変動し、この結果、成膜室内の酸素分圧が変動することとなり、安定した膜性能を得ることが難しいという問題点を有していた。たとえば、ＩＴＯの成膜では、成膜室内の酸素分圧が変動すると、成膜された膜の比抵抗や透過率が変動することが広く知られている。また、ＭｇＯの成膜では成膜室内の酸素分圧が変動すると、成膜された膜の結晶化度や結晶方位が変動することも知られている。特に、圧力勾配型プラズマガンを利用して酸化物蒸着材料が設置されたるつぼを動かしながら長時間の成膜を成膜室内に一定量の酸素を供給し、かつ、成膜室圧力の制御を行なわず実施する場合においては、成膜室内部の酸素量の変動が著しいため、図２に示すように成膜室内の酸素分圧

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が安定せず、よって安定した膜性能を得ることが難しいのみならず、図３に示すように成膜室の圧力も著しく変動するために、成膜速度も大きく変動するとの知見を得た。また、成膜中の成膜室圧力が一定になるように、真空ポンプによる排気速度をその排気口側に設けられた圧力制御用バルブの開度を調整することにより制御する場合においても、成膜中の成膜室圧力の変動が酸素分圧の変動のみに起因しないため、図４に示すように成膜室内の酸素分圧が変動することとなり、安定した膜性能を得ることが難しいという問題点を有していた。本発明は前記問題点を解決し、長時間にわたって安定した膜性能を得ることのできる酸化物薄膜の成膜制御法を提供することを目的としている。

【０００４】

【課題を解決するための手段】本発明は前記目的を達成するために、圧力勾配型プラズマガンを使用して基板に酸化薄膜を形成する活性化反応蒸着法において、処理室内の真空度を検出し、この検出値が設定値と等しくなるように処理室からの排気速度を制御する一方、処理室内の酸素分圧を検出し、この検出値が設定値と等しくなるように処理室内への酸素導入量を制御するものである。

【０００５】

【発明の実施の形態】つぎに、本発明の実施の形態を図１にしたがって説明する。図において、成膜装置Ａは、蒸着材料Ｓを加熱蒸発させる第１処理室１と基板を収容する第２処理室１５とからなり、前記第１処理室１の側壁には公知の圧力勾配型プラズマガン２を備え、底部には上部に蒸着材料Ｓを収納したるつぼ３が設置されている。そして、このるつぼ３は下記するフィルム状基板Ｗの搬送方向上流から下流へ、下流から上流へ所定区間を駆動装置４により往復移動する。また、前記第１処理室１内は酸素ガス供給ノズル５が設けられており、第１処理室１内の酸素ガス分圧が質量分析計６で計測され、その酸素ガス分圧信号とあらかじめ設定されている目標酸素ガス分圧指令との差を酸素ガス流量演算器７で計算し、マスフローコントローラ８により第１処理室１内の酸素ガス分圧が所定値となるように前記酸素ガス供給ノズル５から所定量の酸素ガスから第１処理室１内へ制御供給されるようになっている。

【０００６】さらに、第１処理室１内の真空度は真空計９により計測され、この真空度信号は圧力演算器１０に入力され、ここであらかじめ設定されている目標真空度指令と比較され、その差にもとづき圧力制御バルブ１１が作動して第１処理室１内の真空度を所定値に制御するようになっている。

【０００７】前記第２処理室１５は前記第１処理室１の上方に開口１ａを介して設けたもので、この第２処理室１５の内部にはフィルム状基板Ｗの送り出しロール１６と巻き取りロール１７が配設されるとともに、両ロール１６，１７間にヒータ１８が配設されている。

【０００８】以上の構成において、前記圧力勾配型プラズマガン２を作動させてプラズマビームＰをるつぼ３の蒸着材料Ｓに集束させる。るつぼ３は長時間の成膜に適応できるように多量の蒸着材料Ｓが収納されており、駆動装置４によってフィルム状基板Ｗの搬送進行方向上流から下流へ、下流から上流へと連続的に所定区間往復移動する。そして前記プラズマビームＰで加熱・蒸発した蒸着材料Ｓはこの過程で自身の酸素を一部離脱させながら酸素ガス供給ノズル５から供給される酸素ガスとも反応し、フィルム状基板Ｗに酸化膜を形成する。この際、質量分析計６は前記第１処理室１内の酸素ガス分圧を常時計測して、その酸素ガス分圧信号を前記酸素ガス流量演算器７に送る。酸素ガス流量演算器７にはあらかじめ設定されている目標酸素ガス分圧指令と質量分析計６からの酸素ガス分圧信号との差を演算して酸素ガス供給量を算出し、これを酸素ガス供給量信号としてマスフローコントローラ８に出力し、当該マスフローコントローラ８はこの酸素ガス供給量信号を受けて、その供給量を酸素ガス供給ノズル５へ安定的に供給する。

【０００９】一方、真空計９は前記第１処理室１内の真空度を常時計測して、その真空度信号を前記圧力演算器１０に送る。当該圧力演算器１０はあらかじめ設定されている目標真空度指令と真空計９からの真空度信号との差を演算してバルブ開度信号を算出し、これを圧力制御バルブ１１に出力する。そして当該圧力制御バルブ１１はこのバルブ開度信号を受けて動作し、前記第１処理室



１内の真空度を目標値に導く。

【００１０】

【発明の効果】以上の説明で明らかなように、本発明によれば、第１処理室内の酸素ガス分圧と真空度を別個に計測して制御するため、酸化物蒸着材料自身や蒸発粒子から離脱する酸素量や成膜室内部の各所に吸着した酸素の成膜中における離脱量が変動しても、成膜中は常に前記第１処理室内の酸素ガス分圧は一定となり、さらに成膜中は常に前記第１処理室内の圧力も一定となるため、成膜速度の変動も発生することなく長時間にわたって安定した膜性能を得ることができる。

【図面の簡単な説明】

【図１】　本発明を実施するための成膜装置の概略図。

【図２】　成膜室での真空度と酸素ガス分圧との関係を示すグラフ。

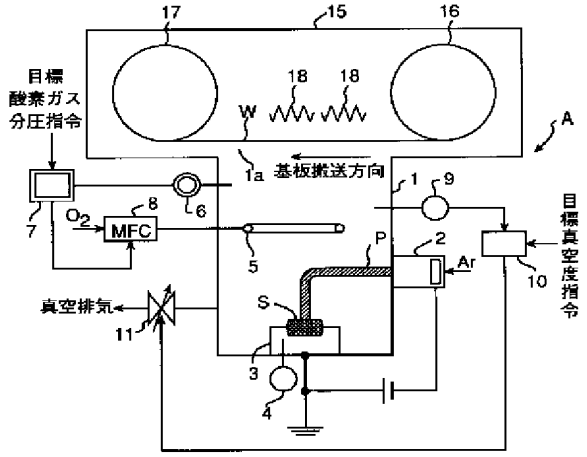
【図３】　成膜室での真空度と成膜速度との関係を示すグラフ。

【図４】　成膜室での真空度と酸素ガス分圧との関係を示すグラフ。

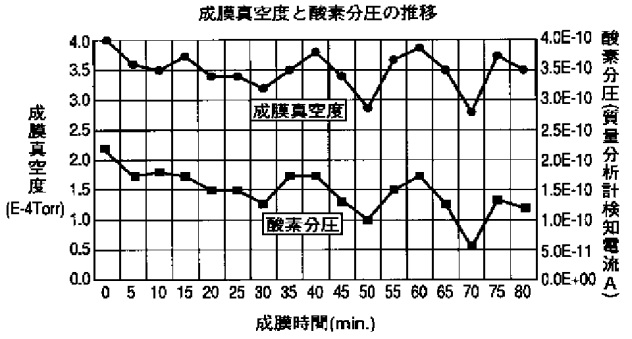
【符号の説明】

１…第１処理室、２…圧力勾配型プラズマガン、３…るつぼ、５…酸素ガス供給ノズル、６…質量分析計、７…酸素ガス流量演算器、９…真空計、１０…圧力演算器、１１…圧力制御バルブ、１５…第２処理室、Ｗ…フィルム状基板。

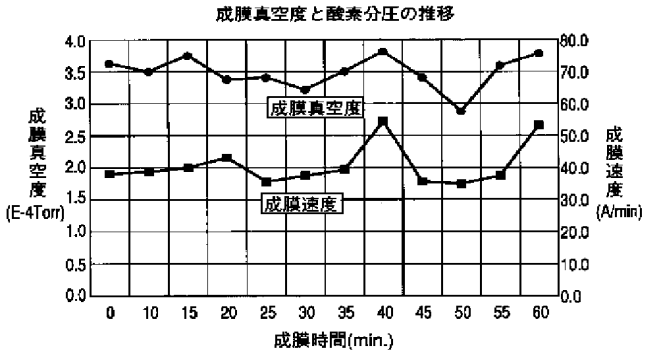
【図１】



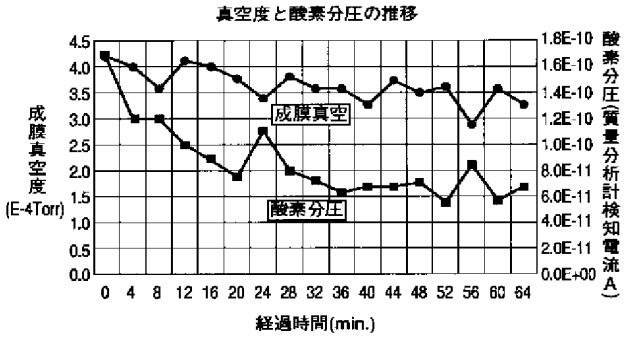

【図２】



【図３】


成膜真空度と酸素分圧の推移
成膜真空度
(E-4Torr)
成膜速度
(A/min)
成膜時間(min.)
成膜真空度
成膜速度

【図４】


真空度と酸素分圧の推移
成膜真空度
(E-4Torr)
酸素分圧(質量分析計検知電流A)
経過時間(min.)
成膜真空
酸素分圧

**DERWENT-ACC-NO:** 1999-267652

**DERWENT-WEEK:** 199923



**TITLE:** Oxide thin film formation controlling method using activated reactive evaporation technique involves maintaining vacuum degree and oxygen partial pressure of processing chamber at preset value, by controlling exhaust speed of film particles and amount of oxygen introduction into chamber

**INVENTOR:** SHINTANI M

**PATENT-ASSIGNEE:** CHUGAI RO KOGYO KAISHA LTD[CHUI]

**PRIORITY-DATA:** 1997JP-245188 (September 10, 1997)

**PATENT-FAMILY:**

| PUB-NO | PUB-DATE | LANGUAGE |
|---|---|---|
| JP 11080952 A | March 26, 1999 | JA |

**APPLICATION-DATA:**

| PUB-NO | APPL-DESCRIPTOR | APPL-NO | APPL-DATE |
|---|---|---|---|
| JP 11080952A | N/A | 1997JP-245188 | September 10, 1997 |

**INT-CL-CURRENT:**

| TYPE | IPC DATE |
|---|---|
| CIPP | C23C14/08 20060101 |
| CIPS | C23C14/54 20060101 |

**ABSTRACTED-PUB-NO:** JP 11080952 A

**BASIC-ABSTRACT:**

NOVELTY - The degree of vacuum of the interior of processing chamber (1) is measured and the exhaust speed of the film particles from the process chamber is controlled so that detected vacuum degree equals a preset value. The amount of oxygen supplied to the process chamber for evaporation of film is controlled so that the oxygen partial pressure equals a preset value.

USE - For controlling formation of thin films such as indium tin oxide film and magnesium oxide film for liquid crystal panel, AC type plasma display.

ADVANTAGE - Property of film is maintained for long time by suppressing the fluctuation of film forming velocity as the pressure of chamber and partial pressure of oxygen is maintained at fixed

rate.

DESCRIPTION OF DRAWING(S) - The drawing shows film forming apparatus. (1) Process chamber.

**CHOSEN-DRAWING:** Dwg.1/1

**TITLE-TERMS:** OXIDE THIN FILM FORMATION CONTROL METHOD ACTIVATE REACT EVAPORATION TECHNIQUE MAINTAIN VACUUM DEGREE OXYGEN PRESSURE PROCESS CHAMBER PRESET VALUE EXHAUST SPEED PARTICLE AMOUNT INTRODUCING

**DERWENT-CLASS:** L03 V05

**CPI-CODES:** L03-G05;

**EPI-CODES:** V05-L05A1;

**SECONDARY-ACC-NO:**

**CPI Secondary Accession Numbers:** 1999-079650

**Non-CPI Secondary Accession Numbers:** 1999-199364